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पेट्रोलियम और उसके उत्पादों की परीक्षण पद्धतियाँ

[पी : 137]

विमान टरबाइन ईंधन के जल पृथक्करण लक्षण

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 137]

WATER SEPARATION CHARACTERISTICS OF AVIATION TURBINE FUELS

UDC 665·753·12 [621·438] : 629·13 : 543·812

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 1991 Price Group 6

Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 137]

WATER SEPARATION CHARACTERISTICS OF AVIATION TURBINE FUELS

Adapted with permission from ASTM D 2550-85

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1 SCOPE

1.1 This test method is for use in rating the ease with which a fuel will release entrained or emulsified water when passed through a coalescing medium. The method is intended to measure the water separation characteristics of fuel as produced (*see* Note).

NOTE — ASTM Field Method D 3602 uses a small scale portable apparatus to rate this characteristic of fuel.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
170 : 1986	Acetone (*third revision*)
537 : 1967	Toluene, pure, nitration grade (*first revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
2631 : 1976	Isopropyl alcohol (*first revision*)

3 TERMINOLOGY

3.1 Water Separation Index, Modified (WISM)

A numerical rating indicating the ease of separating water from fuel by coalescence.

4 OUTLINE OF THE METHOD

4.1 The WSIM of a fuel is measured with an ASTM-CRC Water Separometer, a device in which a water-fuel emulsion is prepared and metered through a cell containing a standardized glass-fibre coalescer. The cell effluent turbidity due to entrained water, is measured by light transmission through the fuel to a photocell. The output of the photocell is fed to a meter with a 0 to 100 scale, from which the numerical rating of the fuel is read. The higher the number, the more readily does the fuel release water.

5 SIGNIFICANCE AND USE

5.1 The test provides a measure of the presence of surfactant agents in the fuel. It is intended to detect carryover of traces of refinery treating residues in fuel as produced, particularly sulphonates and naphthenates. Field Method D 3602 describes a portable test that can also detect surfactant agents acquired by the fuel during distribution and handling in the field. Some of these surfactants could affect the ability of filter coalescers to separate free water from aviation turbine fuels.

6 APPARATUS

6.1 ASTM CRC Water Separometer[1]

Comprises three functions, emulsion preparation, coalescence, and analysis (*see* Note) as shown in the flow diagram of Fig. 1. A detailed description of the instrument is given in Annex A. Maintenance and trouble-shooting instructions are outlined in Annex B.

NOTE — The ASTM-CRC water Separometer differs from the standard model CRC water Separometer in having an extended range rotameter, a separate emulsification loop, and a reduced arc precision coalescer cell[2].

6.2 Hypodermic Syringe

A 2 ml hypodermic syringe with a 300 ml long square-end needle having an inside diameter of 0·30 mm is required. A 1 ml Tuberculin Precision syringe with 1/100 divisions is required for tests with reference fluids.

7 REAGENTS AND MATERIALS

a) *Coalescer Disks*[3]

A new set of standardized coalescer disks (coarse and fine) is required for each test.

b) *Hydrocarbon Flushing Fluid*

A surfactant-free, clean, hydrocarbon material, such as a water-white, solids-free, kerosine-type fluid with a known WSIM of 98 to 100 is suitable. Reference fluid base may be used.

---

[1] The Separometer is available from Emcee Electronics, Inc., 520 Cypress Ave, Venice, FL 34291. Other manufacturers may be approved, but testing laboratories should not purchase testing units except from Emcee Electronics Inc., without ascertaining whether such units have been approved .

[2] A kit of modification parts to convert a standard model CRC Water Separometer to the ASTM-CRC model is available from Emcee Electronics, Inc.

[3] Standardized coalescer disks are available from Emcee Electronics, Inc.

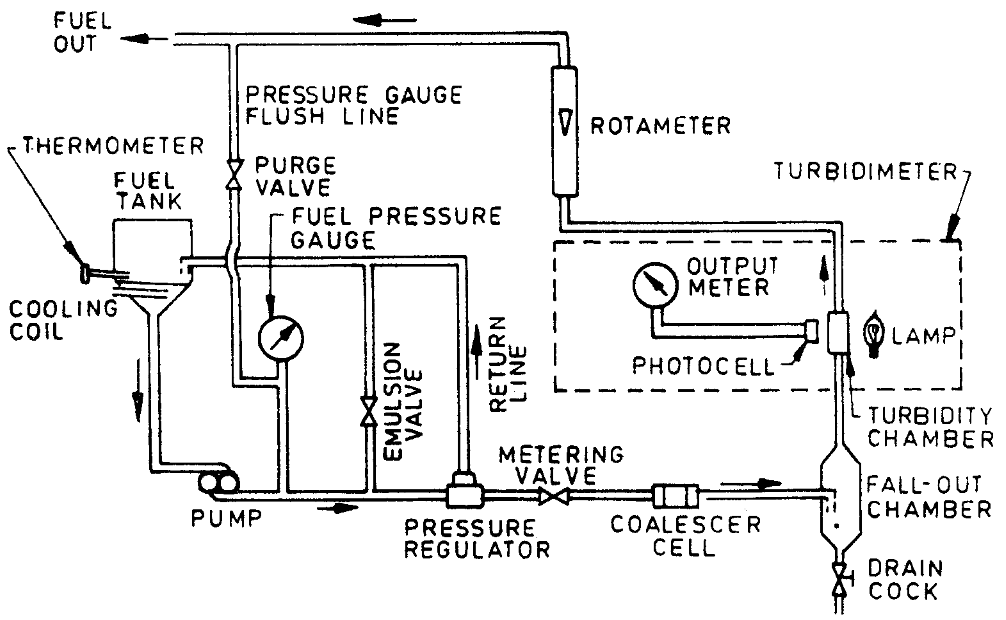


FIG. 1 SEPAROMETER FLOW DIAGRAM

c) *Reference Fluid Base*
A surfactant-free, clean hydrocarban material prepared in the manner described in Annex C.

d) *Isopropanol*
(*see* IS 2631 : 1976).

e) *Acetone*
(*see* IS 170 : 1986).

f) *Toluene*
(*see* IS 537 : 1967).

g) *Dispersing Agents*
A toluene solution containing 1 mg of solid (100 percent dry) bis (2-ethylhexyl) sodium sulphosuccinate/ml of toluene.

NOTE — Aerosol 0T (100 percent) sold by Fisher Scientific Co. Pitsburgh PA, has been found satisfactory for this purpose.

h) *Distilled Water*
(*see* IS 1070 : 1977).

j) *Reference Fluids*
(For checking the condition of the water separometer instrumentation), consisting of various concentrations 0 to 0·8 ml/l of dispersing agent in reference fluid base. The water separation characteristics for this range of concentrations are shown in Table 1. The reference fluids shall be tested in the manner described in Annex D. Results shall fall within the range of limits shown in Table 1 or shall be discarded and the reference fluid check repeated.

Table 1 Expected Performance with Reference Fluids

Concentration of Dispersing Agent, ml/l	Standard Rating	Limits for Acceptable Performance	
		Minimum	Maximum
0	99	97	100
0·2	89	82	94
0·4	80	69	88
0·6	72	59	83
0·8	65	51	77

NOTE — 95 percent confidence level based on results of eight laboratories.

8 PREPARATION OF APPARATUS

8.1 The following procedures shall be adhered strictly to minimize test variability. Deviation from them will assure serious disagreement among laboratories.

8.1.1 *Flowmeter Calibration*

If the rotameter has not been calibrated for the next fuel to be tested, proceed with the calibration before cleaning the instrument. If the calibration is known, or if the instrument is to be shut down, proceed with the cleaning as shown in **8.1.2**. If the instrument is already clean and calibration is not needed, continue with preparation of the coalescer cell (*see* **8.1.3**).

8.1.1.1 To calibrate, first drain the system accorde ing to **8.1.2.3**. Add 250 ml of test fuel to th- tank, start the pump, and circulate one minute (be sure the emulsion valve is closed); then drain the system again according to **8.1.2.3**.

8.1.1.2 Add 600 ml of test fuel at 21 to 27°C to the tank, start pump, and circulate one minute (emulsion valve closed); reconnect the jumper line. Open the emulsion valve to 138 to 207 kPa on the gauge, then open the metering valve to expel air. After large bubbles cease in jumper line, close the emulsion valve and continue to expel air from the outlet system. When the rotameter is free of air, adjust to a float reading of about 20. Measure the time for 100 ml to flow from fuel outlet into a graduated cylinder or other calibrated vessel. Repeat the measurement using float levels of about 16 to 24. Stop the pump. Plot the float levels against the time for 100 ml and draw the best fit straight line. Select the float level which yields a flow of 100 ml in 40 seconds.

8.1.2 *Cleaning*

Clean the apparatus soon after a preceding test or calibration. Cleaning shall be accomplished in the following manner.

8.1.2.1 *Isopropanol wash*

The purpose of this step is to remove water and adsorbed surfactants from the instrument. Drain the system in accordance with **8.1.2.3**. Add 200 ml of isopropanol. Add another 50 ml from a squeeze bottle using the stream to wash down the sides of the tank. (Any convenient source of pressure may be used to produce the stream). Be sure to wash down the entire inner surface of the tank. Start the pump with all valves closed. Allow the fluid to recirculate through the return line for 2 minutes. Open the emulsion valve until the pressure gauge reads 552 kPa and allow the fluid to recirculate for 2 minutes. Close the emulsion valve and open the purge valve for 5 seconds dropping pressure to about 552 kPa. Close the purge valve and drain the system as in **8.1.2.3**. Repeat the foregoing three times (a total of four washes), using a total of 1 litre of isopropanol.

8.1.2.2 *Hydrocarbon flush*

The purpose of this flush is to remove isopropanol. Using the same procedure as with the isopropanol, flush out the system thoroughly with 1 litre of hydrocarbon flushing fluid (a total of four flushes). If the apparatus is to shut down after the last flush, pump the fluid out of the tank without vacuum, but when the tank is nearly empty, shut off the metering valve and turn off the pump leaving the tubing system full of the fluid up to the metering valve. If another test is to follow, drain as shown in the next section.

8.1.2.3 *Draining*

Use this procedure at the end of the test, after each washing or flushing step, and at start-up to remove fluid left in the apparatus at shut-down. Follow the specified order to avoid contaminating the pressure system with fluid from the outlet system. Install a plastic tubing jumper line in place of the coalescer cell. Open the metering valve wide and start the pump. When large bubbles appear in the jumper line, stop the pump.

Caution — Prolonging the operation of the pump dry could damage it. Remove the jumper line, and drain the fall-out chamber. Apply vacuum to the fuel outlet line to draw the residual fluid into a receiver. Open the purge valve wide. After the outlet system is clear (about 30 s), block off the fall-out chamber inlet and close the drain cock to direct the full vacuum to the pressure system. Run the pump 5 seconds. Finally open and close the emulsion valve twice. Then close all valves and disconnect the vacuum.

Caution — When refilling the system purge the air from the gauge line immediately on starting the pump and before pressure has built up. This should be done with negligible loss of fluid.

8.1.3 *Coalescer Cell*

Disassemble the coalescer cell, rinse out the cell parts with a stream of acetone, and dry thoroughly. Apply vacuum to the fittings, not the orifices, to remove excess acetone. Install a set of new coalescer disks in accordance with the diagram of Fig. 2 and reassemble (*see* Note). Install the coalescer cell in the apparatus.

NOTE — Test results can be affected greatly by the amount the coalescer disks are compressed in assembly. The assembly shall be made so that the "compression gap" (crush on the filter) is 1·588 ± 0·013 mm. The gap can be checked in accordance with the dimensions shown in Fig. 2. The coalescer cell can be optionally equipped with quick-disconnect fittings, but surfactant-containing grease or elastomers shall be avoided. Any such fittings shall be easy to clean and dry completely.

8.1.4 *Pump-Output*

The pump output shall be checked periodically. Refer to **B-1.1** for the procedure and output requirements.

9 SAMPLE PREPARATION

9.1 Under no circumstances should the sample be prefiltered after collection. Filtration media can remove surfactants, the detection of which is one of the purposes of this test method. If the test fuel sample is contaminated with particulate matter, allow it to settle before testing.

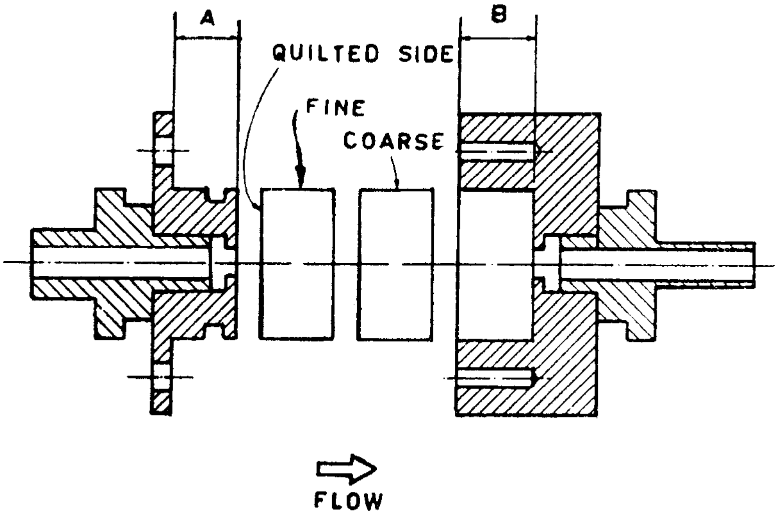
9.2 Special precautions concerning sample containers and sampling technique are given in Annex E (*see* Note).

NOTE — Test method results are known to be sensitive to trace contamination from sampling containers.

10 PROCEDURE

10.1 Warmup

If the apparatus has been turned off following completion of the previous test, turn on the main power switch at least 15 min prior to the start of the flow operation to allow the electronic components to come to equilibrium.



$B—A = 0{\cdot}062\,5 \pm 0{\cdot}000\,5$

FIG. 2 COALESCER CELL

10.2 Checkout

Make sure that the apparatus has been cleaned properly and flushed thoroughly with flushing fluids, as described in **8.1.2**, prior to introduction of the test fuel.

10.3 Test Fuel Flush

A major purpose of these flushes is to standardize the surfaces of the instrument with any surfactant which may be in the test fuel. It is, therefore, of extreme importance that the procedure be followed strictly. Flush twice with the test fuel (Note 1) in the following manner. Introduce 350 ± 5 ml to the fuel tank and turn on the pump. Open the emulsion valve so as to recirculate fuel to the tank for 552 kPa gauge. Close the emulsion valve and open the metering valve to fill the system. In succession, open and close the metering valve several times to rid the system of bubbles insofar as possible. Open the purge valve wide for 3 seconds then close. Adjust the pressure regulator, if necessary, to 689 kPa, with all valves closed. Allow the remainder of the test fuel to flow through the system at the correct float level (100 ml in 40 s) found during calibration. Check the flow rate using a previously calibrated 100 ml graduate or vessel. Continue flowing until the tank is nearly empty. Then close the metering valve and shut off the pump. The time for 100 ml should be 40 ± 0·8 s. If the time falls outside this range during the first test fuel flush, an adjustment may be attempted during the second. Add 250 ± 5 ml of test fuel and proceed with the second flush and calibration check. If the time is still not correct, discard the coalescer disk, repeat the rotameter calibration (*see* **8.1.1.2**) and repeat the cleaning procedure (*see* **8.1.2**). If the calibration is all right, set the output meter to read 100 (*see* Note 2) by means of the adjusting knob. When the tank is nearly empty, shut off the pump and close the metering valve.

NOTES

1 Some fuels might be prone to create a hazard from electrostatic charge buildup, particularly in the fuel tank and waste disposal sections. This hazard may be reduced by operating the system with the tank cover in place and eliminated by employing a nitrogen blanket over the fuel tank and waste disposal system. An appropriate line is built into the Separometer which may be connected to a nitrogen source to provide blanketing over the fuel tank. Since waste disposal system will vary, each laboratory will have to assess hazards and take steps that might be necessary to eliminate them.

2 Experience has shown that the light bulb begins to emit light of wavering intensity long before it burns out. This is especially true if the bulb or instrument has been jarred. A faulty bulb, evidenced by instability in the output meter reading, should be replaced. Occasionally, unstable readings may also be caused by tarnishing of the electrical contacts in the light-control rheostat or in the light bulb socket. The condition may be corrected by "dressing up" the contacts with a piece of fine emery paper.

10.4 Emulsion Formation

Introduce 2 litres of test fuel into the tank and turn on the pump. Open the emulsion valve until the pressure drops to 552 kPa. Make sure that the pressure remains between 538 to 565 kPa for the entire emulsion making period, adjusting the emulsion valve as needed. Using the long-needle hypodermic syringe, introduce 2·0 ml of distilled water at a uniform rate over a period 60 ± 15 s directly into the opening of the line leading from the bottom of the tank. Replace the tank cover. Note the time of start of water injection and allow the mixture to continue to recirculate until a total of 5 minutes has elapsed.

10.5 Test Run

Immediately after the 5 minutes recirculation period, close the emulsion valve and then open the metering valve to allow to the mixture to flow through the system at 150 ml/min. Record the output meter readings at 2 minutes intervals until 1 200 ml of fuel has been processed. Maintain the tank temperature at 24 to 27°C throughout the run. If cooling is necessary, use cooling water only slightly below the test temperature to avoid overcooling and creating excessive thermal gradients.

11 REPORT

11.1 Report as the WSIM the meter reading obtained at the end of 8 minutes after flow starts through the coalescer cell.

12 PRECISION

12.1 Results of duplicate tests shall not differ by more than the following amounts:

Repeatibility	*Reproducibility*
See Fig. 3	*See* Fig. 3

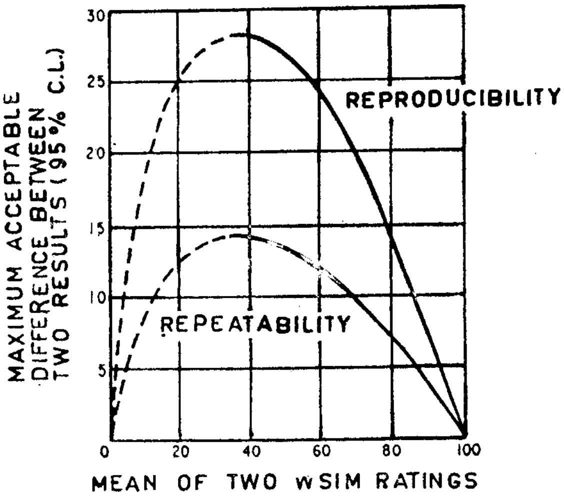


FIG. 3 VARIATION OF REPEATABILITY AND REPRODUCIBILITY WITH RATING LEVEL

ANNEX A

(*Clause* 6.1)

ASTM-CRC WATER SEPAROMETER

A-1 APPARATUS

A-1.1 The apparatus described shall be known as the ASTM-CRC Water Separometer, Model 1104, as modified by Severity Kit 1104 k4. The automatic features provided by 1104 A, B and C are permissible.

A-1.2 All essential instruments and parts of the Water Separometer itself are furnished with the unit. A list of parts for the unit may be obtained from Emcee Electronics, Inc. (*see* Note under **B-1.1** for replacement pumps).

A-2 GENERAL DESCRIPTION

A-2.1 The ASTM-CRC Water Separometer is shown schematically in Fig. 1 and is illustrated.

A-3 FUEL SYSTEM

A-3.1 The Separometer fuel system consists of the following major components and their associated piping and electrical connections, fuel tank and cover, fuel pump, emulsion valve, metering valve, pressure regulator, rotameter, coalescer cell, water fallout chamber, and turbidimeter. Operation of the fuel system is best described by tracing the fuel path from the tank to final disposal.

A-3.1.1 *Pump and Metering Valve*

Fuel flows by gravity from the tank to the pump inlet. Pump outlet pressure is maintained by regulator which returns excess fuel back to the tank. Flow of fuel from the regulator to the rest of the system is controlled by the metering valve. Fuel returned to the tank is heated by passage through the pump. A water cooling jacket is provided to maintain constant temperature of fuel in the tank.

A-3.1.2 *Emulsion Valve*

The valve is used only during emulsion preparation and only with the metering valve closed. Under these conditions, the pressure regulator does not control, and pressure is manually set with the emulsion valve. This valve is closed, after the emulsion is prepared, for the remainder of the test.

A-3.1.3 *Coalescer Cell and Water-Fallout Chamber*

The coalescer cell receives the fuel-water emulsion from the metering valve and causes some portion of the water droplets to agglomerate. The coalescer cell has an orifice diameter of 3·55 ± 0·013 mm. These are given opportunity to settle in the fallout chamber.

A-3.1.4 *Turbidimeter*

Fuel passes upward from the fallout chamber to the turbidity chamber. Here, light of standardized intensity passes through the fuel to a photocell. The light reaching the photocell is governed by the amount scattered or absorbed by water droplets in the fuel. Current output of the photocell appears on the output meter, mounted on the front panel.

A-3.1.5 *Rotameter*

From the turbidity chamber, fuel passes through the rotameter where flow rate is measured. Fuel is then discharged overboard to waste disposal.

A-3.1.6 *Fuel Sytem Accessories*

The purge valve, located on the left side of the cabinet and above the utility connections is used to flush out the pressure gauge piping loop between tests. It is closed at all other times.

A-4 ELECTRICAL AND VENTILATING SYSTEM

A-4.1 Refer to Fig. 4 for a schematic diagram of the electrical system. The location of each electrical control is described below.

A-4.1.1 *Power Switch*

Power to the unit is controlled by the power switch located on the front panel.

A-4.1.2 *Pump Switch*

The pump is controlled by the pump switch located on the front panel.

A-4.1.3 *Output Meter*

The meter is located on the front panel and reads the output of the photocel in the instrument section. The reading of this meter is the WSIM, taken 8 minutes after flow starts through the coalescer cell.

A-4.1.4 *Output Meter Adjust*

This is a rheostat whose control knob is located on the front panel just below the meter. It is used to set light intensity during the test fuel flush so that the meter reads 100.

A-4.1.5 *Fuse*

A 4-Amp slow-blow fuse is located just below the power switch on the front panel.

A-4.1.6 *Blower Motor*

This motor is located inside, the cabinet near the right side. It runs as long as the power switch is turned "ON".

A-4.1.7 *Instrument Lamp*

This lamp is mounted on the access door at the right side of the cabinet.

A-5 INSTALLATION

A-5.1 Service Requirements

A-5.1.1 *Power*

A three-wire cord supplies power to the Separometer through a female socker located low on the left side of the cabinet. Normally, the unit is supplied to operate on 115-V, 60-Hz current. Options available are 220-V, single phase, 50-Hz current, and constant voltage where fluctuation is more than ±15 V options can be had singly or in combination.

A-5.1.2 *Water*

This is required to control fuel temperature. Tubing connections are located on the left side of the cabinet. A valve to control flow is needed on the inlet side only.

A-5.1.3 *Nitrogen*

This is optional but recommended (*see* Note under **8.1.3**). A tubing connection is provided on the left side of the cabinet.

A-5.1.4 *Waste Fuel Disposal*

The fuel-out connection shall be provided with a tube leading to a drain can or to a sink leading to a sewer approved for receiving hydrocarbon.

A-5.1.5 *Space Requirements*

Overall height : 660 mm
Overall width : 966 mm
Overall depth : 356 mm
Weight : 79·4 kg
Bench area : 0·47 m², *Min*
Height of bench : optional
Overhead clearance : 610 mm, *Min* (recommended)
Right side clearance : 305 mm, *Min*
Left side clearance : 152 mm, *Min*
Rear clearance : none required

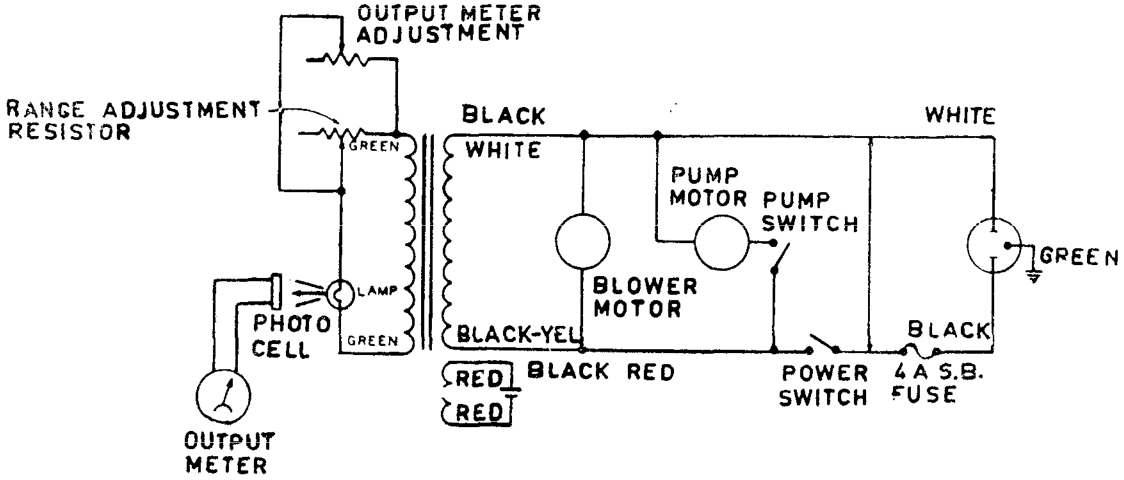


FIG. 4 SEPAROMETER WIRING DIAGRAM

ANNEX B

(*Clause* 6.1)

MAINTENANCE

B-1 CALIBRATION AND ADJUSTMENT

B-1.1 Pump

The pump delivery should be 415±15 ml/min determined at 689 kPa by measuring the return flow to the reservoir with the metering valve, emulsion valve, and purge valve closed. Reference Fluids, described in 7 (c) (for a fuel of similar viscosity) are used at a fuel temperature of 24 to 70°C. This delivery should be checked every 100 runs. The pump should be replaced when the flow drops below 400 ml/min.

NOTE — Replacement pumps meeting the delivery requirement of 415±15 ml/min at 680 kPa offered for use in the ASTM/CRC Water Separometer are as follows:

LFE (Eastern) Model 1201-198 is available from ERDCO Engineering Corp., P.O. Box 1310. Evanston. IL 60204.

LFE (Eastern) Model 1231-LBG-332 (McEmcee Part No. 104-00-5335) is available from McEmcee Electronics 8875 Midnight Pass Rd., Sarasota. FL 33581.

The precision of this test method was developed using ASTM-CRC Water Separometers with pumps of the type LFE (Eastern) Model 1201-198. The precision expressed in Section 11 may not be valid when using Water Separometers with type LFE (Eastern) 1231-LPG-332 pumps.

B-1.2 Pressure Regulator

The pressure regulator shall be adjusted as necessary to 689 kPa. To do this, remove the cover at the top of the pressure regulator, loosen the lock nut, and turn the screw until the desired pressure is obtained (The pump shall be running while making this adjustment).

B-1.3 Turbidimeter Range Resistor

A variable resistor located in the rear of the cabinet is used to adjust the sensitivity and range of the turbidimeter. This is factory set; but, if the meter cannot be set to 100 by the output meter adjust knob, the adjustment of the slide on the range resistor will correct this.

B-1.4 Calibration

Calibrate the fuel thermometer and pressure gauge initially and when checking the condition of the separometer.

B-1.4.1 To calibrate the fuel thermometer, remove it and immerse it in a bath at 24°C together with a standardized glass thermometer.

B-1.4.2 To calibrate the pressure gauge, inset a standard gauge in place of the coalescer cell. Add fuel to the tank and operate the pump with all valves closed except the metering valve. Adjust the pressure regulator until 689 kPa is indicated on the standard gauge. If necessary mark the pressure gauge to indicate 689 kPa.

B-2 PERIODIC MAINTENANCE

B-2.1 Fall-Out Chamber

Periodic cleaning of the glass with chromic acid followed by copious rinsing with distilled water followed by drying with reagent grade acetone is recommended.

B-2.2 Turbidity Chamber

Clean as with fall-out chamber.

B-2.3 Lubrication

Fifty-hertz models require periodic lubrication of the speed changer assembly at the three bearing locations.

B-3 TROUBLE SHOOTING

B-3.1 Table 2 gives a list of possible malfunctions which may be encountered together with possible causes and remedies.

Table 2 Possible Malfunctions and Remedies

(*Clause* B-3.1)

Sl No.	Malfunction	Probable Cause	Corrective Action
1.	No power to until	Blown fuse	Replace fuse
		Failure of power source	Restore service
2.	Pump fails to work	No power to unit	*See* item 1
		Pump switch broken	Replace switch
		Faulty wiring	Repair wiring
		Worn out motor	Replace motor
3.	Pump delivery rate low	Worn pump	Replace pump
		Motor malfunction	Replace motor
		Dirt or obstruction in lines	Clean out lines
		Crimped tubing	Replace tubing
4.	Incorrect pressure	Loose lock nut in pressure regulator	Reset pressure, tighten lock nut
5.	Excessive pressure drop during test	Loose packing nut on metering valve	Tighten packing nut
6.	Desired pressure cannot be obtained	Broken diaphragm of broken spring in pressure regulator	Replace or repair pressure regulator
		Fuel pressure gauge needle struck	Dry off front cover of dial and bend needle to move freely
		Faulty pump	*See* item 1
		Dirt or obstruction in tubing	Clean or replace tubing
7.	Output meter does not function	Burnt-out bulb	Replace bulb
		Needle struck	Tap meter gently
		Faulty wiring	Repair wiring
		Faulty photocell	Replace photocell
8.	Fluctuation of output meter	Worn bulb	Replace bulb
		Pitted contact points	Dress contact points in bulb socket, on bulb and on rheostat
		Excessive power fluctuations	Install constant voltage regulator between power source and water separometer
9.	Failure to get 100 output on meter with output meter adjust knob	Worn bulb	Replace bulb
		Pitted contact points	Dress contact points in bulb socket, on bulb and on rheostat
		Incorrect voltage to bulb	Adjust auxiliary variable resistor with control knob set at 50
		Worn photocell	Replace photocell
		Dirty turbidimeter cell	Clean cell
		Excessively dark-coloured fuel	Output meter reading of 100 cannot be obtained with excessively dark fuel. This is not a normal problem in evaluating jet fuels.
10.	Malfunction of rotameter float	Dirt in tube	Clean tube
11.	Fuel will not flow through rating system of unit	Dirt in tube	Clean tube
		Struck rotameter float	Clean rotameter tube
12.	Failure in check calibration curve Fig. 2	Extraneous contamination in refence fluid	Extreme care shall be exercised in fluid handling to prevent contamination. All containers, glassware, etc, especially new ones, should be cleaned with suitable solvents to remove traces of detergents, flux, oils and other surface active substances

ANNEX C

[*Clause* 7 (c)]

PREPARATION OF REFERENCE FLUID BASE

C-1 SCOPE

C-1.1 This procedure describes the preparation within an 8 hour day of a 20 L lot of reference fluid base. The procedure has been found to give a filtrate having 100 WSIM rating.

C-2 SUMMARY OF PROCEDURE

C-2.1 A fuel is flowed at a constant rate through a fresh column of granular clay and collected in a clean storage receiver. The fuel should conform to relevant IS Specification and the aromatic content should be between 10 and 20 volume percent.

C-3 APPARATUS

C-3.1 Glass Column

Containing a sealed-in coarse fritted glass disk near the bottom and with a 4 mm metering type TFE-fluorocarbon stopcock outlet at the bottom. The inside diameter of the column in 55 to 66 mm and the length above the fritted disk shall be at least 1 m.

C-3.2 Siphon

Of glass tubing having an inside diameter of 5 to 10 mm with the legs 100 to 150 mm apart. The suction leg shall be 380 to 400 mm long to reach the bottom of the feed container and shall be bevelled at 45°C. The other leg shall be 50 to 100 mm longer and shall bent at a right angle 10 to 200 mm from the end.

C-3.3 Feed Container

Shall be any standard square or round 20 litre can in which the sample is obtained.

C-3.4 Receiver Can

Shall be a new 20 litre epoxy lined can or one which has been used only with clay-filtered fuel. Plastic containers shall not be used.

C-3.5 Metal Funnel

With a 10 to 20 mm outlet.

C-3.6 Graduated Cylinder

Of 0·5 to 1 litre capacity.

C-3.7 Graduated Cylinder

Of 50 to 100 ml capacity.

C-3.8 Beaker

2 litre capacity.

C-4 MATERIALS

C-4.1 Attapulgus Clay

30/60 mesh, LVM (calcined) grade or equal. Store the clay protected from atmospheric moisture and avoid handling that will cause particle size segregation.

C-4.2 Fine Glass Wool

C-4.3 Reagent Chloroform in a Squeeze Bottle

C-5 PREPARATION OF APPARATUS

C-5.1 Mount the column vertically (*see* **C-6.1** and **C-6.5** as to matching location to feed and receiver cans).

C-5.2 Measure 500 ml of clay in the graduated cylinder, tapping gently to settle.

C-5.3 Place the funnel atop the column with its outlet centered. Open the stopcock wide. Quickly up-end the cylinder of clay placing its outlet directly into the funnel. Aim the funned so that the clay falls exactly in the centre of the column. Remove the funnel and tap the column gently all round to settle and level the clay bed. Tamp a fist-sized wad of glass wool carefully down on top of the bed.

C-6 FILTRATION PROCEDURE

C-6.1 Position a full 20 litre feed container with its opening level with the top of the column. Prop up the side of the container opposite the opening 20 to 30 mm. Remove the cap and insert the siphon, short leg in the can, long leg in the column.

C-6.2 Support the 2 litre beaker under the column.

C-6.3 Make sure the column stopcock is wide open. Put slight air pressure on the feed can to start the siphon. The glass wool packing should prevent the clay bed being disturbed at startup (*see* Note).

NOTE — In a well-prepared column, the fuel may be seen to advance down the column in a nearly horizontal plane; and no bubbles will rise through the clay. If the advancing front is tilted more than 45° or there is much bubbling, the quality of the percolation may be impaired.

C-6.4 As soon as the fuel is flowing through the column outlet, adjust the metering screw to a rate of 50 to 55 ml/min. Check by measuring for 1 or 2 min intervals with the small graduated cylinder.

C-6.5 When at least 1 litre has been collected, turn off the stopcock without disturbing the metering screw setting. Remove the beaker and support the 20 litre receiver can under the column so that the outlet tube extends about 10 mm into the opening. Open the stopcock. Protect the opening from dirt (*see* Note).

NOTE — Caution — When percolating flammable fuel, seal between the outlet and receiver opening with aluminium foil, ground the receiver, and purge it with dry nitrogen before starting flow into it. A similar purge of the column before the step in C-6.3 is desirable.

C-6.6 Recycle the beaker or filtrate to the feed can or discard it.

C-6.7 When the level of fuel has dropped nearly to the top of the clay bed, turn off the stopcock, remove and cap the receiver can (*see* Note).

NOTE — At the specified flow rate, the 20 litre percolation will be completed in 6 to 6½ h running time. If necessary, the rate may be increased to a maximum of 60 ml/min.

C-6.8 For lengthy storage, the receiver can should be dry nitrogen purged.

C-7 CLEANING THE COLUMN

C-7.1 Drain the column.

C-7.2 Dismount the column, up-end it over a solid waste can; and with the stopcock wide open, blow out the clay.

C-7.3 With the column inverted over a liquid waste receiver, run chloroform from the squeeze bottle into the outlet. Tilt the column to rinse the entire disk area and the entire inside of the column. When the clay residue has been entirely rinsed out, disassemble and rinse the stopcock parts, dry and reassemble them, and blow the entire assembly dry.

C-7.4 If the column still appears dirty, rinse it thoroughly with hot water, then distilled water. Invert it and rinse as in **C-7.3** with acetone, then chloroform, and blow dry. This should seldom be necessary.

ANNEX D

[*Clause* 7 (J)]

PROCEDURE FOR TEST WITH REFERENCE FLUIDS

D-1 Prepare a 20 litre lot of reference fluid base in accordance with Annex C.

D-2 Prepare a 100-ml quantity of dispersing agent in accordance with **7**(g).

D-3 Test with reference fluid base neat.

D-3.1 Test the reference fluid base neat in the same manner as any test fuel. Follow procedures in accordance with Sections 8, 9, 10 and 11.

D-3.2 Results shall either meet the criteria of Table 1 or shall be discarded and the reference fluid base check repeated.

D-3.3 If repeated checks of the reference fluid base fail to meet the criteria given in Table 1, discard the reference base and prepare a new one. It is permissible to retreat off test fluid in accordance with Annex C.

D-4 TESTS WITH REFERENCE FLUIDS

D-4.1 Prepare blends of the dispersing agent and the reference fuel base in the separometer fuel tank.

D-4.2 Follow procedures in accordance with Sections 8, 9 up to and including **10.2**. Paragraph **10.3** shall be ignored and the following procedure followed.

D-5 PREPARATION OF REFERENCE FLUIDS

D-5.1 Introduce 2 500 ml (exactly) of neat reference fluid base into the separometer tank. Turn on the pump. Open the emulsion valve until the pressure drops to 551 kPa. Make sure that the pressure remains between 537 to 565 kPa for the entire additive blending period.

D-5.2 A 1-ml Tuberculin Precision syringe with 1/100 divisions or equivalent shall be used with long needle to introduce the desired quantity of dispersing agent directly into the opening of the line leading from the bottom of the tank. Table 3 summarises the required injection volumes and rates of injection.

Table 3 Dispersing Agent Injection Volumes

Concentration of Dispersing Agent, ml/l		Minimum Time for Injection of Dispersing Agent, Seconds
0·2	0·5	30
0·4	1·0	60
0·6	1·5[1)]	60 and 30[1)]
0·8	2·0[1)]	60 and 60[1)]

[1)] Two injections are necessary for each test to obtain the total volume indicated.

D-5.3 Note the time of start of dispersing agent injection and allow the mixture to recirculate until a total of 15 min has elapsed.

D-5.4 Test Fuel Flush

A major purpose of these flushes is to standardize the surfaces of the instrument with the dispersing agent. It is, therefore, of extreme importance that the procedure be followed strictly. Flush twice with the test fuel in the following manner. With the emulsion valve closed, open the metering valve to fill the system. In succession, open and close the metering valve several times to rid the system of bubbles insofar as possible. Open the purge valve for 3 s then close. Adjust pressure to 689 kPa if necessary. Allow fuel to flow through the system at the correct float level (100 ml in 40 s) found during calibration until a total of 250±5 ml has been collected. Then close the metering valve. During this time check the flow rate with a calibrated 100-ml graduate. The time for 100 ml should be 40±0·8. If the time falls outside this range, an adjustment may be attempted during the second flush. Open the metering valve and proceed with the second fuel flush, calibration, and meter reading adjustment. Collect exactly 250±5 ml, of fuel and then close the metering valve. If the calibration is satisfactory, set the output meter to read 100 by means of the adjusting knob while fuel is flowing and before the 250-ml quantity has been collected.

D-5.5 The test fuel is now in the separometer tank; and the test should proceed with emulsion formation, *see* **10.4** (*see* Note).

NOTE — The purpose of preparing the test blends containing dispersing agent in the separometer is to minimize errors that might be caused by adsorption of the additive on surfaces of another blending container. Some data have been presented showing that WSIM ratings sometimes improve significantly with time when the fuel sample is placed in scrupulously clean containers.

D-5.6 Duplicate results shall either meet the repeatability criteria of Table 1 or shall be discarded and the reference fluid check repeated.

ANNEX E

(*Clause* 9.2)

SAMPLING TECHNIQUE

E-1 For any test that seeks the presence of trace constituents, steps could be taken to assure testing a representative sample. The round-robin precision study for this method of test showed that the flushing of the instrument was most important. This indicates that the trace amounts of surfactant material in aviation turbine fuels can be adsorbed on, or desorbed from, metal surfaces. A suggested technique for taking Separometer samples follows. It has been found to give representative samples. Any similar approach should be satisfactory. The technique is shown here only as a guide to good practice.

E-1.1 Sample Container

Epoxy-lined containers are preferred.

E-1.2 Sample Source

The sample should be drawn from a moving stream of fuel whose source shall be removed from tank water bottoms by as great a distance as feasible.

E-1.3 Sample Line

The line may consist of a short one/fourth to 6·4 to 12·7 mm diameter tube with its open end facing the moving stream. The other end (outside the pipe) should be equipped with a suitable shutoff valve and spout.

E-1.4 Taking the Sample

First, flush the sample line with 3·8 litres of the fuel to be sampled. Second, rinse the sample can with three separate 0·95 litre amounts of the fuel to be sampled for 3·8 litres can. Include the cap, and inner seal if used in the rinsing. Third, draw the sample and put the cap in place.

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